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Paths of Development of Organic Derivatives of Phosphorus.

(Presented at Plenary Meeting of Conference).

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There are many examples in the history of development of organic chemistry when some section of this science, which previously had not attracted much attention of the scientists, begins to develop vigorously owing to an unexpected practical application of representatives of that class of compounds. An example of this is the chemistry of organosilicon compounds.

At the beginning of this century silicon, as the closest analog of carbon, attracted a considerable amount of scientific attention. It seemed that it would be possible to try to create on the basis of silicon a region of a chemistry as extensive as that of organic chemistry. However it soon appeared that the creation of chemistry of silicon is impossible and the interest in organic derivatives of silicon declined considerably.

In the 40's of this century, owing mainly to the work of K.A.Andrianov, it appeared that on the base of organic derivatives of silicon it is possible to realize the synthesis of many new materials which are valuable for industry (lacquers, plastics, lubricants, synthetic rubber with unusual properties). After this the chemistry of organic derivatives of silicon entered a period of vigorous development. There were many interesting discoveries of not only practical significance but also of purely theoretical character.

Another example.- Discovery of sulfa preparations as valuable medicinals led to enhanced development of this region of organic chemistry. In a short period there were synthesized over 5000 new sulfamide preparations. And the discovery of cancerogenic properties of polynuclear hydrocarbons led to a rapid growth of chemistry of polynuclear compounds.

At this time the chemistry of organic derivatives of phosphorus is living

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Through such a period of vigorous development, occasioned to a considerable degree by the fact that organic derivatives of phosphorus were found to be unexpectedly applicable to the most diverse branches of the national economy.

However it should be noted that this intensive development of any area of science, and specifically organic chemistry, is possible only because during the previous period there had been prepared by the work of many scientists a solid theoretical and experimental foundation for it.

In this presentation I wish insofar as possible to outline the various stages of development of chemistry of organic derivatives of phosphorus and to show the significance which has been presently acquired by organic derivatives of phosphorus in the chemical industry, agriculture and other branches of the national economy as well as in medicine.

The organic derivatives of phosphorus are known for over a hundred years. However phosphorus attracted attention of investigators to a considerably lesser degree that did its closest neighbors in the D.I.Mendeleev periodic system- arsenic and particularly nitrogen.

There is no point to speak here of the significance of nitrogen-bearing organic compounds (it is known that proteins belong to this class of organic substances).

Organoarsenic compounds attracted a considerable amount of attention after the discovery of Salvarsan and its use as a medicinal. Preparations containing arsenic began to be used widely in the medical practice. Later, intensive development of the chemistry of organic derivatives of arsenic was closely connected with the application of some of them as war-poison substances.

The insufficient attention paid to the chemistry of organophosphorus compounds is manifested by the fact that there are several monographs which are devoted to arsenic and antimony, while until 1940, when the book by V.M. Rodionov "Organic compounds of phosphorus" was published, there was not a single monograph on organophosphorus compounds.

I shall pause briefly on the history of development of the chemistry of

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phosphorus compounds.

In 1846 Thenard synthesized methyl-, dimethyl- and trimethylphosphine. These organophosphorus compounds ignited in air and had strongly poisonous properties. Owing to these unpleasant properties Thenard did not study them further.

In 1854 Railton prepared by the action of phosphorus trichloride on alcohol what he supposed to be esters of phosphorous acid. However, it appeared later that Railton had a mixture of substances.

The period of 1854-1874 several workers prepared and studied some organophosphorus compounds. However these studies had the occasional and unsystematic character.

Beginning with 1874 and until 1915, i.e over forty years, the study of organophosphorus compounds was done by Michaelis and his school in Germany. Michaelis and his students prepared and described a tremendous number of organophosphorus compounds belonging mainly to the aromatic series, and developed a number of synthetic methods preserved significantly up to the present time.

In 1905 A.E.Arbuzov published his dissertation devoted to the preparation of neutral esters of phosphorous acid. He showed that all the previous workers did not have pure specimens of esters of phosphorous acid and he also showed the reasons because of which the workers could not obtain these substances in the pure state. A.E.Arbuzov opened the way of transformation of neutral esters of phosphorous acid containing trivalent phosphorus into esters of alkylphosphonic acids - derivatives of pentavalent phosphorus. This transformation, known under the name of Arbuzov rearrangement, is one of the main methods of synthesis of esters of phosphonic acids.

In 1914 A.E.Arbuzov published his second dissertation in which he explained in more detail the phenomenon of the Arbuzov rearrangement with examples of both neutral esters of phosphorous acid as well as esters of phenylphosphorous and diphenylphosphinous acids.

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Approximately at the same time organic derivatives of phosphorus were being studied by Kipping and Meisenheimer from the viewpoint of possible existence of optically active phosphorus.

From P.S.Pishchimuka, a student of A.E.Arbuzov in New Alexandria, came in 1911 a large piece of work on the esters of thiophosphoric and selenophosphoric acids and their derivatives.

As it is evident from these very brief data the studies of this period had a purely theoretical character and no one of the workers in the field of organic derivatives of phosphorus could even suppose that in the near future these studies would be of interest not only to scientists but also to the industrial workers. At the same time, as I shall attempt to show later, the work by A.E.Arbuzov on rearrangement of esters of phosphorous acid which occurs under the influence of halogen derivatives and hydrogen halogen acids remains the foundation for many processes which at this time are realized on plant scale while the work of P.S.Pishchimuka serves as the foundation for preparation of a number of organic derivatives of phosphorus used and proposed for use in various areas of industry and agriculture.

In 1917 Polish scientists Milobendzkii and Sakhmowskii developed a method of preparation of esters of phosphorous acid by the action of alcohols on phosphorus trichloride in the presence of tertiary amines (pyridine) which bind the hydrogen chloride evolved in the reaction. This work, not noted at that time, later played a great role in the technological formulation of a series of reactions of preparation of organic derivatives of phosphorus.

It is necessary to note the studies by the American chemist James Conant (1924-1930) of the action of phosphorus trichloride and phenyldichlorophosphine on aldehydes and ketones and the work of Swedish scientist Nylen on the use of the Michaelis-Becker reaction for the synthesis of esters of phosphonic acids.

In 1931 in the laboratory of organic chemistry of Kazan University during studies which had for their purpose the clarification of a purely

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[illegible]ical question about the fate of the phosphorus part in the reaction [illegible]eration of free triphenylmethyl radical according to the method of [illegible] and B.A.Arbuzov, we prepared for the first time esters of pyrophospho[illegible], [illegible]phoric, mono- and dithio-pyrophosphoric acids and isolated in the pure [illegible] this ester of pyrophosphoric acid.

All further studies in the field of organophosphorus compounds also [illegible] the purely scientific, theoretical character.

Thus in the course of nearly ninety years the purpose of studies of [illegible] derivatives of phosphorus was the expansion of own knowledge in the [illegible] chemistry of phosphorus, solution of scientific problems which were important to organic chemistry based on organophosphorus compounds. These [illegible] did not pusue and could not pursue any practical goals.

However it would be incorrect to think that during this period the [illegible]anic derivatives of phosphorus could not find any practical applications. [illegible]ters of phosphoric acid were used as plasticizers for nitrocellulose [illegible] cellulose acetate with lowering of flammability in the articles made [illegible] these materials. Esters of dithiophosphoric acid found a wide area of use [illegible] flotation agents. Dicresyl esters of dithiophosphoric acid and sodium salt [illegible] diethyl ester of dithiophosphoric acid turned out to be excellent flotati[illegible] [illegible] for enrichment of ores of rare metals.

[illegible] this practical application of organic derivatives of phosphorus [illegible] to produce a notable stimulating action on the development of the [illegible]istry of phosphorus, apparently owing to the single type of compounds [illegible] and the relative simplicity of their production. In the work of V.M. [illegible] there are devoted but two pages to the technological significance of [illegible]phosphorus compounds.

It appeared that the development of biochemistry should stimulate the [illegible]pment of the chemistry of organic derivatives of phosphorus. Even at the beginning of the present century it became known thatesters of phosphoric [illegible] play a very important role in the process of transformation of carbo-

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[illegible]ses, in alcoholic fermentation, in glycolysis of muscle tissue and some other processes which take place in living organisms. However even in this [illegible] the very important role in biochemistry of the esters of phosphoric [illegible] failed to affect the development of the synthetic part of the studies [illegible]rganic derivatives of phosphorus.

Another section of biochemistry where esters of phosphoric acid play an important role is devoted to phosphatides. The presence of phosphorus in the brain substance was discovered by Vaucquelin as long ago as 1812. Lecithins, cephalins and substances related to them served as objects for a great number of studies. The section of nucleotides played and still plays a not lesser role in biochemistry, these substances being also connected with phosphoric acid. In the 40's of the present century there was established the presence of the residue of pyrophosphoric acid in adenosine triphosphate which turned the attention to pyrophosphoric acid as a component of physiologically active substances.

However even the development of these important areas of biochemistry failed to exert a strong influence on the chemistry of organic derivatives of phosphorus in the broad sense of the word. Only in the past 10-15 years owing to the work of the English scientist Alexander Todd on the synthesis of nucleotides has this area of organic and biological chemistry shown a close connection with a new direction in the chemistry of phosphorus- with derivatives of pyrophosphoric acid and gave a new direction in the synthesis of organic derivatives of phosphorus.

I have extended somewhat my excursion into the area of biochemistry but I still consider it necessary to mention even briefly the connection between [illegible] derivatives of phosphorus with some enzymes, vitamins and hormones.

In 1935-1938 Euler established the structure of the enzyme cozymase and showed that into its composition there enters the residue of pyrophosphoric acid. Approximately at the same time there was established the structure of [illegible]xylase also containing a residue of pyrophosphoric acid and closely

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...ected with vitamin B_1. The yellow enzyme of Warburg also turned out to ... phosphoric ester.

One could extend the familiarity with the materials on the connection ... important physiologically active compounds with organic derivatives ... phorus. It seems to me that the shown examples testify sufficiently ... significance of organic derivatives of phosphorus in biochemistry ... ology.

At the same time I underline the fact that this connection with biochem... y ... not affect noticeably the development of the chemistry of organic deri... atives of phosphorus.

Extensive works on organic derivatives of phosphorus were conducted ... fore and during the Second World War (Note: not the National War...) ... Germany. However this work became known only after the war since it was ... nected with the military organizations and [redacted] strictly classified.

During the advance by our troops into Eastern Prussia there were d... ... producing the war poison substance Tabun which contains phosphor... ... occupation of Western Germany there fell into the hands of the ... troops the archives of the firm I.G.Farbenindustrie and specifi... ... terials about its plants in Elberfeld and Leverkusen. Especially int... ...ing were the materials on the plant in Elberfeld about new powerful ... ticides which were related to organic derivatives of phosphorus.

In 1947 there was published a [redacted] paper by the former collaborator ... I.G Farbenindustrie Schrader, in which he described the results of the ... then ten-year labor on the search among organic derivatives of phosphor... ... preparations which possess insecticidal action. However these preparations ... used in Germany as insecticides since the military powers placed the se... ...cy on all this work.

...termination of the Second World War it appeared that also in ... ...had been secretly conducted intensive studies headed by Saunders, ...s and others, on the synthesis and study of properties of physiologi...

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organophosphorus compounds.

The work in these two directions- synthesis of insecticides and "OV" (abbreviation for war gases) undoubtedly affected the further development of chemistry of organic compounds of phosphorus. Their effect would have been more noticeable had they not been classified at one time.

Schrader began his work on organophosphorus compounds in 1936. Soon were discovered compounds of the type of mixed anhydride of acetic and the ester of dimethylamidophosphoric acid having strong insecticide action, then the fluoride of the ethyl ester of dimethylamidophosphoric acid, having even stronger action as an insecticide, and other fluorophosphates. All these compounds had contact action.

In 1940 there was prepared a compound of the type of tetramethyldiamido fluorophosphate which possessed not only a contact but also a strong so called systemic action. Plants sprayed with this substance absorbed it and became for several weeks poisonous to insects.

Tests of all these preparations on warm blooded animals showed their very high toxicity. This was one of the reasons why they failed to find at that time any practical application.

A wide study of the physiological activity of organic derivatives of phosphorus permitted the discovery of their myotic action on the eye pupil and later their anticholinesterasic action.

In England and in Germany there was prepared a very active substance diisopropyl fluorophosphate, quite toxic to warmblooded animals. In 1946 it was suggested for treatment of certain eye diseases.

All these studies were accompanied by much work on the search for industrial methods of synthesis of the indicated types of organophosphorus compounds.

Studies on mixed anhydrides naturally led to the thought of trying to test the insecticidal action of esters of the acids of phosphorus with the anhydride character, which was done by Schrader in 1938 with esters of

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[illegible]phosphoric and then thiopyrophosphoric acids.

Tetraethyl pyrophosphate displayed an amazingly strong contact action [illegible] insects (in concentration of 0.001% on aphids) but turned out to be a very powerful poison for warmblooded animals.

Tetraethyl pyrophosphate was prepared for the first time by us in 1931 in the laboratory of organic chemistry in the Kazan University during the studies of purely theoretical character. At that time we synthesized for the first time esters of monothiopyrophosphoric acid, while Schrader prepared esters of dithiopyrophosphoric acid.

At that time we never had a thought that these new compounds are so [illegible] and we worked with them with the precautions which are usual for an [illegible] chemist.

All the esters of pyrophosphoric and thiopyrophosphoric acid indicated by me did not find an application in the practical agriculture until the end of the second World War since they were classified by the German military.

To the derivatives of pyrophosphoric acid belongs one of the most interesting insecticides with systemic action- octamethyltetramide of pyrophosphoric acid (OMPA or Octamethyl).

In concluding the survey of the prewar and war periods of studies in the area of organic derivatives of phosphorus I wish to underline that at the end of the war in Germany there were found and were being manufactured on a large scale the most powerful war gases belonging among organophosphorus compounds. Thus in Germany there was being produced 1000 tons per month of Tabun or Trilon-83 (ethyl ester of dimethylamido-cyanophosphoric acid) and Sarin or Trilon-46 (isopropyl ester of the fluoride of methylphosphonic acid) was being made at 600 ton per month rate.

Publication of these secret materials on the studies of organophosphorus compounds (insecticides, poisonous substances and possible medicinals) aided the development of the studies of organic derivatives of phosphorus.

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At the present time the chemistry of organic derivatives of phosphorus is passing through a period of vigorous growth. Literally in every issue of the main chemical journals of various countries there are papers on organophosphorus compounds. Organic derivatives of phosphorus are being studied not only scientific institutes and laboratories of the institutions of higher learning. Research in the field of organophosphorus compounds is carried in foreign countries also to industrial companies and every year there are obtained hundreds of patents on preparation and use of organic derivatives of phosphorus for the most diversified uses. For example in the index of the reference journal Chemical Abstracts for 1954 in the section on phosphorus compounds alone there are references to over 2000 papers and patents.

The most intense work is going on in the area of preparation of new substances with insecticidal action. Great value exists in mixed esters of thiophosphoric acid among which are E605, Thiofos or Parathion, Metacide, Systox as well as Malathion.

In the foundation for this tremendous synthetic operation there lie the works of the pioneers of chemistry of organophosphorus compounds Michaelis, A.E.Arbuzov and other scientists.

Even at the present time the theoretical studies are opening the paths not only for synthesis of new preparations but also for the realization of production of some of them in large plant scale.

There are many examples in organic chemistry in which rare substances which had been prepared in the laboratory in amounts of few tens of grams began to be produced in tens or many hundreds of thousands of tons after they had found an important practical use. This was true of diisopropyl ether, butadiene which until the work of the deceased Academician S.V.Lebedev was being laboriously prepared in the laboratory in amounts of tens or hundreds of grams, while now in the world production this amount is expressed by a million tons.

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In the chemistry of organophosphorus compounds there are also being [illegible] new paths which assure the preparation in large amounts of substances which only a short time ago appeared to be inaccessible and exotic. As one of such examples one may cite esters of pyrophosphoric and thiopyrophosphoric acids. In 1931 we isolated with much difficulty from a complex mixture of substances the esters of subphosphoric and pyrophosphorous acid and by addition of sulfur to them prepared the previously unknown classes of organophosphorus compounds- esters of monothio- and pyrophosphoric acid. Ester of pyrophosphorous acid was prepared by us in the yield of 1.6% and we had four grams of it. Esters of subphosphoric acid were availbale to us in the amount of twenty grams. Now in the Kazan Section of The Academy of Sciences SSSR for the investigative purposes we prepare in the laboratory up to 70 kilograms of the ester of thiopyrophosphoric acid per year, and up to 50 kilograms of the ester of dithiopyrophosphoric acid.

Soviet chemists have occupied a leading place in the development of the chemistry of organic derivatives of phosphorus. They preserve this leading place even at this time in the chemistry of organophosphorus compounds. While not long ago Kazan was the single large center in which the chemistry of organic derivatives of phosphorus was being developed, now in our land there are several large centers, which develop successfully this interesting area of organic chemistry.

I should like to give in the most general terms a conception of what new material has been introduced into the chemistry of organophosphorus compounds, what new paths of synthesis of organic compounds have been proposed recently and what has been the role of Soviet chemists in this work.

One of the fundamental methods of synthesis of esters of various phosphoric acids is the Arbuzov rearrangement. By means of this reaction there has been prepared in recent times a large number of new compounds. At the same time it was found that this rearrangement does not always proceed normally and that it produces some interesting deviations. Thus, M.I.Kabachnik, and then

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rov and K.V.Nikonorov found that in the union of esters of thio-
sph ds and thiophosphonous acids with alkyl halides the reaction may
anomalously. N.P.Bogonostseva (A.M.Butlerov Institute at the Kazan
sity) discovered a new direction for the reaction between phosphites
halogen derivatives, which lead to reduction of the halogen compo
of the most interesting anomalous courses of the Arbuzov rearrangement
the formation of unsaturated esters of phosphoric acid discovered for the
time by Perkow in Germany in 1952, and apparently independently of h
by A.N.Pudovik in Kazan and Allen and Johnson In USA. This anomalous reac
proceeds with haloaldehydes, haloketones and esters of haloacids and leads
to the formation of substances which possess strong physiological action and
insecticidal activity.

Considerable attention has been also devoted to the anomalous course of
the Michaelis-Becker reaction.

A new synthesis of organic derivatives of phosphorus which permits one
to prepare in a short period of time a very great number of most varied
esters of phosphonic acids is the reaction of addition of dialkyl phosphites
to unsaturated compounds discovered by A.N.Pudovik during the work on allyl
rearrangements. N.N.Mel'nikov (Research Institute on Fertilizers and Insect
fungicides in Moscow) carried this reaction over to the acid esters of di-
thiophosphoric acids and in this way prepared a large number of preparations
with powerful insecticidal action.

The indicated paths of synthesis of organic compounds of phosphorus found
the expression in numerous patents abroad.

A new synthesis of esters of alpha-hydroxyphosphonic acids is the reaction
of dialkyl phosphites with aldehydes and ketones discovered for the first
time by A.E.Arbuzov and M.M.Azanovskaya and widely extended in the work of
V. Abramov who discovered the reaction of aldehydes with phosphites.

In 1955 Schrader proved the possibility of passage from alpha-hydroxy
phosphonic esters to esters of phosphoric acid. By means of this reaction

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...me possible to synthesize powerful insecticides.

M.I.Kabachnik and his coworkers in USSR and Fields in USA combined the reaction of aldehydes and ketones with dialkyl phosphites with the action of ammonia, which led to a new simple synthesis of esters of alpha-amino phosphonic acids.

In the work on A.N.Nesmeyanov and K.N.Anisimov there received a wide development the method of addition to unsaturated compounds on the part of phosphorus pentachloride which had been used by Bergman and Bondi in Germany in 1931.

I shall indicate also the rather interesting studies by A.V.Kirsanov who opened a new page in the chemistry of organophosphorus compounds.

Also of interest are the studies on addition of tertiary phosphines to double bonds and polymerization of olefins under the influence of tertiary phosphines (Horner, Federated German Republic) which opened new possibilities of application of organophosphorus compounds for both purely scientific and practical purposes.

I do not have the opportunity to list many very interesting studies published in recent years both in Soviet and foreign journals.

The synthetic possibilities in the field of organic derivatives of phosphorus were greatly expanded by these new paths and modifications of the old. Many of them made available for production some organic derivatives of phosphorus on a large scale. As an example of this I shall mention the chlorination of dialkyl phosphites which leads to chlorides of dialkyl phosphates, preparation of pyrophosphates by hydrolysis of the chlorides, etc.

As it is evident from the cited material, the theoretical work is closely connected with the problems of great applied significance. The close interweaving of theory and practice is especially well shown at this time in the area of organophosphorus compounds. Studies which apparently had only the completely detached character, literally within a year or two become important for the solution of purely practical problems.

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Organic derivatives of phosphorus present a very interesting and valuable material for the study and the solution of fundamental problems of modern organic chemistry. One of such problems is that of tautomerism of organic compounds. The problem of tautomerism of organic derivatives of phosphorus had long stood and now stands in the center of attention of Soviet investigators, both organic and physical chemists. I shall note the rather interesting work in this direction by M.I.Kabachnik in Moscow and A.I.Brodskii in Kiev. I shall also indicate that we in Kazan are devoting considerable attention to this problem as well. And in this case also the theoretical studies turn out to be important for practice since the structure of the compounds thus prepared often depends on this phenomenon of tautomerism. In turn the structure reflects sharply on the physiological activity of the compounds.

Here are some of the areas in which organic derivatives of phosphorus have found or may find soon a practical application:

1) ore flotation
2) solvents and plasticizers
3) nonflammable plastics
4) insecticides
5) valuable lubricating oil additives
6) polymerization processes
7) medical preparations

In my report I mentioned only in passing the application of organic compounds of phosphorus as medicinal preparations. Some of these are already being used in treatment of glaucoma, as for instance Mintakol (Fosfakol), and the substance Armin of A.I.Razumov. Organophosphorus will be probably useful in various paralyses-pareses. I think that this is just the beginning of the application of derivatives of phosphorus in medicine and that organophosphorus compounds contain great and unexpected possibilities.

It is not accidental that to this conference came representatives of various scientific disciplines, various branches of national economy.

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[illegible] along with chemists there are present entomologists, botanists, veterinar[illegible] physiologists, pharmacologists and medics. During the sessions we shall hear and discuss the reports of various contents and characters. These various [illegible] express it seems to me the clear showing as to how the field of [illegible]rganic derivatives of phosphorus is being expanded and how deeply it is [illegible]ating the neighboring sciences and the various branches of national [illegible]my, how necessary is the friendship of chemists with representatives [illegible]ther sciences, the close contact of scientists with production workers. I [illegible]re that this contact will solidify further and that it will give [illegible] valuable material for both science and the national economy.

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